HIGH-STRENGTH FIBER-OPTIC WAVEGUIDE

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June 1979

MDA-903-78-C-0289

Quarterly Report 3

For period 1 November 1978 through 31 January 1979

ARPA Order No. 3591

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Arlington, VA 22209

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ARPA Order Number	3591
Name of Contractor	Hughes Research Laboratories
Effective Date of Contract	1 May 1978
Contract Expiration Date	31 July 1979
Contract Number	MDA 903-78-C-0289
Name and Phone Number of Principal Investigator	G.D. Robertson 456-6411, ext. 300
Short Title of Work	High-Strength Fiber-Optic Waveguide
Contract Period Covered by this Report	1 November 1978 - 31 January 1979

This research was sponsored by the Defense Advanced Research Projects Agency under ARPA Order No. 3591, Contract No. MDA 903-78-C-0289.

UNCLASSIFIED

SECURITY CLASSIFICATION OF THIS PAGE (When Data Entered)

REPORT DOCUMENTATION PAGE		READ INSTRUCTIONS BEFORE COMPLETING FORM
1. REPORT NUMBER	2. GOVT ACCESSION NO. AD-A148 562	3. RECIPIENT'S CATALOG NUMBER
4. TITLE (and Subtitle) HIGH-STRENGTH FIBER-OPTIC WAVEGUIDE		5. TYPE OF REPORT & PERIOD COVERED Quarterly Report No. 3 1 Nov 1978 - 31 Jan 1979
		6. PERFORMING ORG. REPORT NUMBER
7. AUTHOR(s) G.D. Robertson		8. CONTRACT OR GRANT NUMBER(s) MDA 903-78-C-0289
9. PERFORMING ORGANIZATION NAME AND ADDRESS Hughes Research Laboratories 3011 Malibu Canyon Road Malibu, CA 90265		10. PROGRAM ELEMENT, PROJECT, TASK AREA & WORK UNIT NUMBERS ARPA Order No. 3591 Program Code 8D10 Prog. Element Code 61101E
11. CONTROLLING OFFICE NAME AND ADDRESS Defense Advanced Research Projects Agcy (DoD) 1400 Wilson Boulevard Arlington, VA 22209		12. REPORT DATE June 1979
		13. NUMBER OF PAGES 25
14. MONITORING AGENCY NAME & ADDRESS(if different from Controlling Office)		15. SECURITY CLASS. (of this report) UNCLASSIFIED
		15a. DECLASSIFICATION DOWNGRADING SCHEDULE

16. DISTRIBUTION STATEMENT (of this Report)

Approved for public release; distribution unlimited.

17. DISTRIBUTION STATEMENT (of the abstract entered in Block 20, if different from Report)

18. SUPPLEMENTARY NOTES

19. KEY WORDS (Continue on reverse side if necessary and identify by block number)

High-strength silica fibers
Fiber-optic waveguides
Metal-clad fibers
Static fatique of silica

20. ABSTRACT (Continue on reverse side if necessary and identify by block number)

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A series of Al-clad fibers to which plastic overcoats had been commercially applied were evaluated. For those resin coatings that involved a high-temperature cure, fiber strength was significantly lower. This fact, coupled with strength reductions obtained when the Al jacket was applied at higher-than-normal temperatures, strongly suggests that a chemical reaction may be occurring between the Al jacket and the surface of the silica fiber.

A preliminary experiment to explore the effects of furnace drawing conditions resulted in a wide spread in the strength distributions of Al-clad fibers. Although further work is needed to identify all of the key process variables, this experiment demonstrates that it is possible to prepare Al-clad fiber with a mean tensile strength of about 600 kpsi in a carbon muffle furnace. This is an improvement of $\sim$200 kpsi over the strength of the standard "good" fiber that we have produced during the past two years.

Evaluation of the new specimen configuration has been almost completed in the static fatigue program being conducted at UCLA. The results are very encouraging and techniques for sample preparation now appear adequate to avoid premature failure of the outer surface of the specimen. Initial testing of doped glass samples with internal oblate bubbles is scheduled for the next quarter.

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SUMMARY

In this quarter (the third) of the program, we continued to investigate a variety of processing parameters that affect both the strength and optical characteristics of Al-clad silica. We also (on a complementary IR&D effort) relocated our drawing facility to new and enlarged quarters and activated a second drawing apparatus. This new equipment includes an induction-heated drawing furnace that can be operated with oxidizing, neutral, or reducing atmospheres in the hot zone. Because this rearrangement in facilities has temporarily reduced our drawing capacity, we have not yet drawn fibers from all-synthetic-glass preforms or waveguides with high numerical aperture. Both types of preforms have been prepared, and drawing work with them will begin soon.

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SECTION 1

INTRODUCTION

The goal of this program is to develop procedures for fabricating continuous 10-km lengths of low-loss fiber-optical waveguide that will exhibit survival probabilities in excess of 95% at 2% strain for 10-year service in high-humidity environments. The effort centers around a unique procedure developed at HRL for hermetically sealing the glass fiber in a metallic jacket. This hermetic jacket provides substantial protection to the glass fiber surface from the deleterious effects of moisture and other environmental contaminants that cause static fatigue (i.e., a reduction in fiber strength with time). Test results on recently prepared glass fibers evaluated in a humid environment under high stress indicate that survival times are at least five orders of magnitude longer for the Hughes aluminum-coated fibers than for state-of-the-art polymer-coated fibers. Details of this development effort are presented in the first quarterly report for this program. #1

To provide the hermetic seal of the glass fiber surface, we have adapted a freeze-coating technique that had been explored by others many years ago for a variety of structural materials applications. Through very careful attention to details, we have been able to apply thin, defect-free layers of aluminum and other metals to silica fibers at drawing rates up to about 2 m/sec. Although the ultimate tensile strength achievable with these metal-clad fibers has not yet equaled that of the best plastic-clad fibers, their resistances to static fatigue — as evidenced by both static and dynamic test methods — is orders of magnitude superior to that of the best plastic-clad fibers. For applications involving fiber stress in deployment or usage, the metal-clad fiber system offers a unique advantage.

Early calculations had suggested that the presence of the metal cladding ought to contribute only negligibly to optical attenuation by absorption of energy in the evanescent fields of guided modes. However,

experience soon revealed that the difference in attenuation of similar waveguides with and without a metal jacket varied widely and was much greater than could possibly be attributed to energy absorption by the metal. These excess losses now appear to be caused primarily by microbending somehow induced by the metal jacket. The previous report presented the first comprehensive set of data on how these excess losses behave when the waveguide is subjected to axial strain. The results suggest, but do not clearly define, several mechanisms that may be at work and also point to means for reducing both the inherent and strain-induced excess loss caused by the metal jacket.

Section 2 presents the results of current program efforts to improve both the optical and mechanical characteristics of metal-clad optical fibers.

SECTION 2

ACCOMPLISHMENTS

A. STRENGTH DISTRIBUTION OF Al-CLAD SILICA

During this quarter we evaluated the strength of Al-clad fibers to which different plastic overcoats had been applied by commercial wire-coating companies. The objective was to investigate both the mechanical and corrosion protection that might be given to the Al jacket by a thin plastic overcoat. Two slightly different epoxy coatings were applied by electrostatic precipitation of finely divided powder onto the Al jacket by the Profile Plastics Co. (Glendale, California). The coating was then subjected to a low-temperature (150°C) oven cycle for 2 to 3 min to fuse the particles into a dense film. Also, four commercial coatings were applied at California Fine Wire Co. (Grover City, California) by dip coating and subsequent evaporation of the solvent in a high-temperature oven ($\sim$340 to 430°C). The oven time-temperature cycles are proprietary, so we have only an *approximate* idea of the high-temperature exposure of the Al-clad fiber during the processing steps. One of the four resins (a polyurethane) did not wet the Al surface and hence did not form a continuous film. The other three all gave coatings of good appearance. These resins were a polyvinyl formal (Formvar), a polyester (Isonel), and a polyimide (Pyre ML).

The strength of the Al-clad fiber was characterized before and after the coating operations by our standard tensile evaluation at a strain rate of 20%/min. For each sample, 25 specimens of 0.5-m gauge length were pulled to rupture; the results were plotted against the cumulative hazard function, as explained in Quarterly Report #1. When we retested the plastic-overcoated samples, which had all shown very similar strength distributions before the overcoating operation, we found essentially no change for the two epoxy coatings or the Formvar. However, there was a large decrease in strength for both the Isonel and

the Pyre ML coatings, which are known to require a higher curing temperature than the other systems. In Figure 1, curve A is for the uncoated specimens and for the epoxy and Formvar coated samples. Curves B and C show the degradation experienced by the Isonel and Pyre ML systems. We currently suspect that a chemical reaction between the Al jacket and the silica fiber surface is responsible for this loss in strength. At high temperatures, the reaction

$$4\ Al + 3\ SiO_2 \rightarrow 2\ A_2O_3 + 3\ Si$$

proceeds readily. A thorough search of the literature has not revealed any kinetic data for this reaction that are directly applicable to the Al-clad silica fiber system. We are now considering a set of experiments to determine the actual rate of reaction and its temperature dependence so that we can define a safe operational time-temperature envelope for the Al-clad fiber.

Along these lines, we have also observed a change in the initial strength distribution of Al-clad silica when the temperature of the molten Al in the coating tip is changed. When operating well, the standard coating process produces a distribution like that already seen for curve A of Figure 1, which is repeated in Figure 2. When, in an attempt to decrease the thickness of the freeze-coated Al jacket, the temperature of the tip was increased by about 40°C, strength was greatly reduced, as shown in Figure 2. The most likely cause of this degradation is an increased chemical attack on the SiO_2 surface at the higher temperature, which produces larger surface defects (Griffith-type flaws). We believe this effect is related to the degradation noted above for the plastic coating processes and expect that high-temperature aging studies should provide a better understanding of the reaction kinetics.

It has been appreciated for some time that a variety of factors can affect the strength distribution of metal-clad fibers. In addition

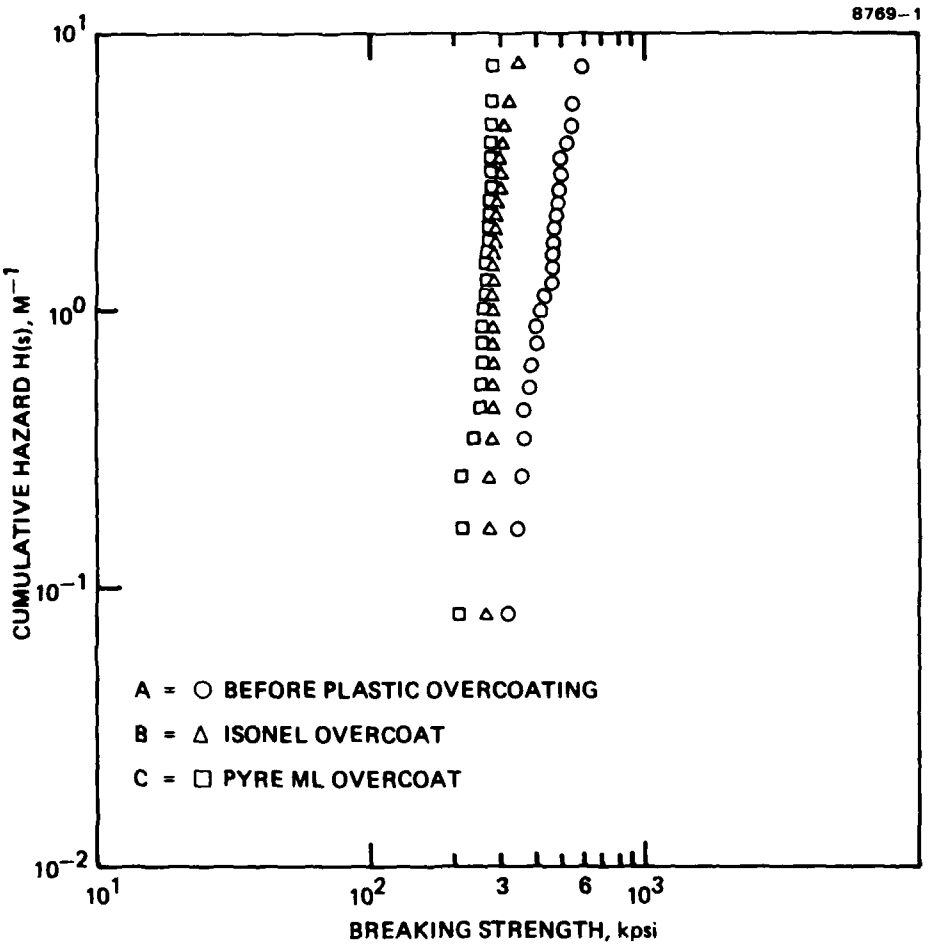

Figure 1. Effects of high-temperature cure on Al-clad silica fibers. Curve A represents the original fiber and curves B and C show the strength deterioration after the application of Isonel and Pyre ML coatings.

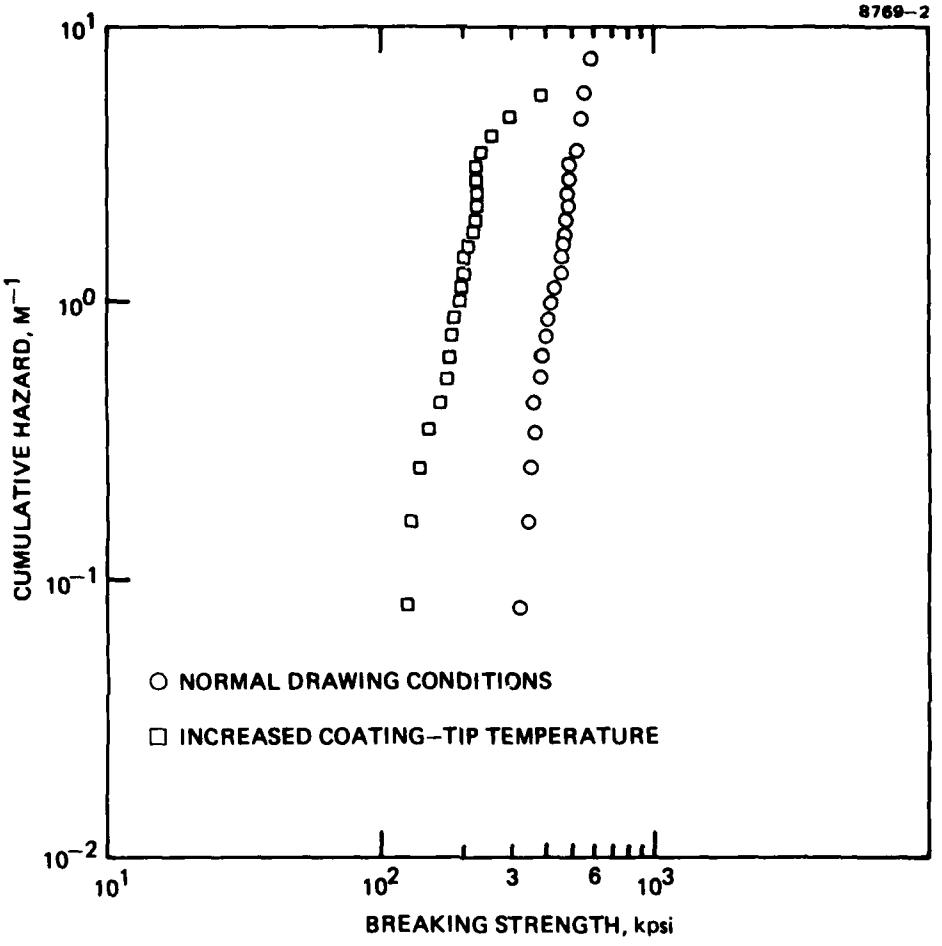

Figure 2. Effect of Al coating tip temperature on strength. An increase of 40°C in the temperature of the molten Al in the coating tip is responsible for the significant drop in strength.

to the temperature effects already noted, we have been exploring the influence of the furnace environment. At present, we are drawing inside a carbon muffle tube protected by a nitrogen atmosphere. There are adjustable water-cooled diaphragms and N_2 gas inlets both at the top and at the bottom of the carbon muffle to control the gas flow in the hot zone. We expect differences in the cleanliness of the hot zone (located about halfway between the diaphragms) when the gas flow rates and diaphragm openings are altered. To avoid temperature fluctuations in the drawing zone caused by the chimney effect, the upper diaphragm is always closed as tightly as possible around the preform without actually touching it. For this set of experiments, the upper opening was 12 mm in diameter, which is just slightly larger than the Suprasil rod preforms.

The variable furnace parameters consisted of adjustments to the gas inlet streams at the top and bottom of the Astro furnace as well as to the size of the opening in the lower diaphragm. The effects of these changes were assessed by comparing the tensile strength distributions of Al-clad fibers drawn under each condition. The effects of furnace gas flow caused a wide range of strength distributions. Table 1 lists the furnace conditions and the median strengths for the resulting tensile strength distributions, which are shown in Figure 3. Also listed in Table 1 for convenience are the minimum and maximum strengths observed for each set of 25 specimens. The distributions for samples 1 and 4 are nearly identical, except for the onset of a pronounced "low-end tail" for sample 4. This type of distribution is typical of both the carbon and the zirconia muffles during the past two years. Occasionally, the maximum and median strengths are higher (as for example the data given in Figures 1 and 2), but these represent the very best obtained under the standard drawing conditions.

The distributions shown for samples 2 and 3 are thought to result from gas flow conditions that produce cleaner or dirtier gas environments around the preform at and above the drawing zone. The results obtained

Table 1. Results of Furnace Parameter Alterations

Sample No.	Diaphragm Opening, mm		Gas Flow, liters/min		Strength, kpsi			Preform Tip Condition after Drawing
	Top	Bottom	Top	Bottom	Median	Max	Min	
1	12	6	6.7	2.5	323	373	241	dirty
2	12	4	0	8.0	587	677	217	clean
3	12	6	[a]	6.7	160	372	107	dirty
4	12	6	2.5	6.7	340	424	76	clean

[a]Gas partially exhausted through the upper port, flow rate unknown.

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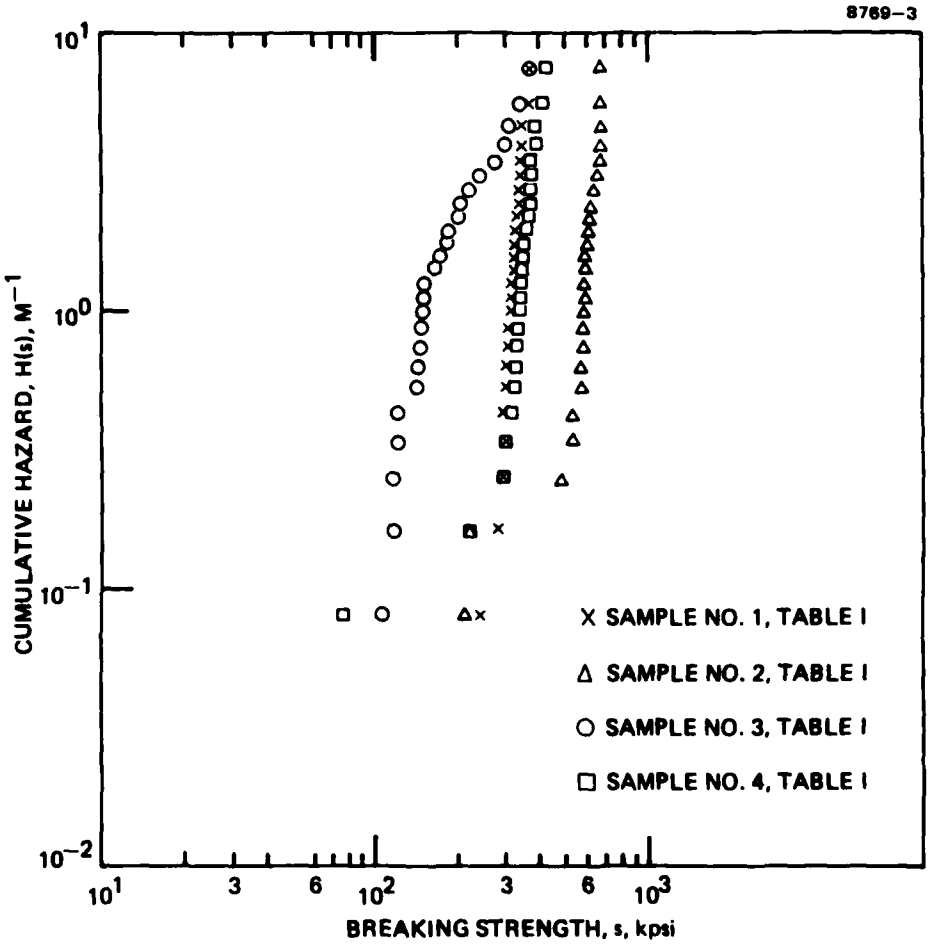

Figure 3. Strength distributions for various furnace modifications. The strength deterioration is attributed mainly to contamination of the preform because of improper gas flow within the drawing region. The results for Sample No. 2 are the best yet seen for Al-clad SiO_2.

with sample 2, which are the best yet obtained for Al-clad fibers, are very encouraging because they demonstrate that strong fiber can indeed be prepared in a muffle furnace. As yet, the data are insufficent to define the optimum drawing condition in the furance, but this good distribution indicates that further work with a muffle-type furnace is certainly justified, and there appears to be no fundamental funace limitation that would require a shift to either laser or torch heating sources.

We have not yet adequately characterized the cause of the "low-end tail." This tail is especially evident for samples 2 and 4, but if a longer length of fiber were tested, such tails would undoubtedly also appear for the other samples as well. Previous work has shown that at least some (if not all) of the low-strength breaks occur at openings in the Al jacket. We have examined rupture surfaces from low-strength breaks in a scanning electron microscope but have not yet been able to identify any particular causes of failure. Continued effort is thus needed to identify the causes of these low-strength ruptures and to define the furnace conditions responsible for the high median strength of sample 2.

B. OPTICAL PROPERTIES CONTROL

As discussed in the previous report, one of the more effective ways to reduce excess microbending loss caused by any coating-induced strains is to increase the numerical aperture (NA) of the core, thereby coupling optical modes more tightly to the core region. We have been working with increased concentrations of GeO_2 and P_2O_5 in the glass. To prevent the preform from shattering because of thermal expansion mismatch, very careful control of the thermal gradients in the preform is required during its manufacture. Although we do not yet have all parameters controlled to our satisfaction, we have been able to produce two preforms with an NA of about 0.30, a considerable increase over the nominal 0.22 of those that we have been making for the past two years. After being drawn into unclad waveguides, one of these exhibited an

attenuation of 7.3 dB/km at 0.85 μm (GaAs) and 23.9 dB/km at 0.63 μm (HeNe). The second waveguide showed considerably higher loss (16 dB/km at 0.85 μm and 26 dB/km at 0.63 μm), which may have been caused by excess moisture entering the CVD apparatus when it was reworked between the fabrication of these two preforms. Unfortunately, at the moment, our spectral insertion loss apparatus is not working, and we have not yet evaluated the high-NA waveguides at wavelengths that would clearly reveal the presence of excess OH in the glass. However, these early results are very encouraging and indicate that, with careful attention to fabrication and handling, we can make high-NA preforms that do not self-destruct.

C. ALL-SYNTHETIC-GLASS PREFORMS

As the first step in studying the "low-end tail" of the strength distribution of long lengths of fiber, we have prepared two preforms from which all-synthetic-glass fibers will be drawn. These preforms were prepared by the conventional internal CVD method with an extra thick barrier of B_2O_3-SiO_2 glass as the first layer deposited. This was followed with a conventional GeO_2-SiO_2-P_2O_5 core (nominal NA ≈ 0.22). After the preform had been collapsed to a solid rod, the outer starting jacket of TO-8 fused quartz was dissolved away in an HF solution until all of the original SiO_2 tube was removed. One of the two preforms has been etched and is ready for drawing and coating with Al. The o.d. of this preform is only 0.180 in., considerably smaller than the 0.3-in. to 0.4-in.-diameter range that we normally work with. However, it is large enough to yield several hundred meters of 125-μm-diameter waveguide. The etching of the second preform is being postponed until after test results have been obtained on fiber drawn from the first preform.

D. NEW DRAWING FACILITIES

A major change in our fabrication capabilities for experimental fibers and waveguides occurred during this quarter with the move of our original drawing machine to a new location and the addition of a second drawing apparatus. Although our output has been considerably below normal during this transition period, we expect experimental productivity to increase soon. The second drawing machine is equipped with an induction-heated drawing furnace that can operate with reducing, neutral, or oxidizing atmospheres. As yet, completely satisfactory operation of the furnace has not been achieved. We anticipate that these normal start-up transients will be resolved during the next quarter. Figure 4 shows the new drawing facility after its installation. The tower design is modular (in contrast to our first machine) to permit easy modification as well as disassembly for eventual transport to a future production facility.

E. STATIC FATIGUE STUDIES IN DOPED SILICA

The purpose of the static fatigue investigation being conducted at UCLA is to use the so-called oblate bubble technique to determine the extent of static fatigue in doped fused silica used in optical waveguides. This technique and the unique problems posed by fused silica are described in the Second Quarterly Report, which identified the following tasks:

(1) Developing tensile test procedures

(2) Making suitable specimens

(3) Achieving fracture initiation at the bubble surface.

This section describes results achieved in each of these tasks since the second quarter.

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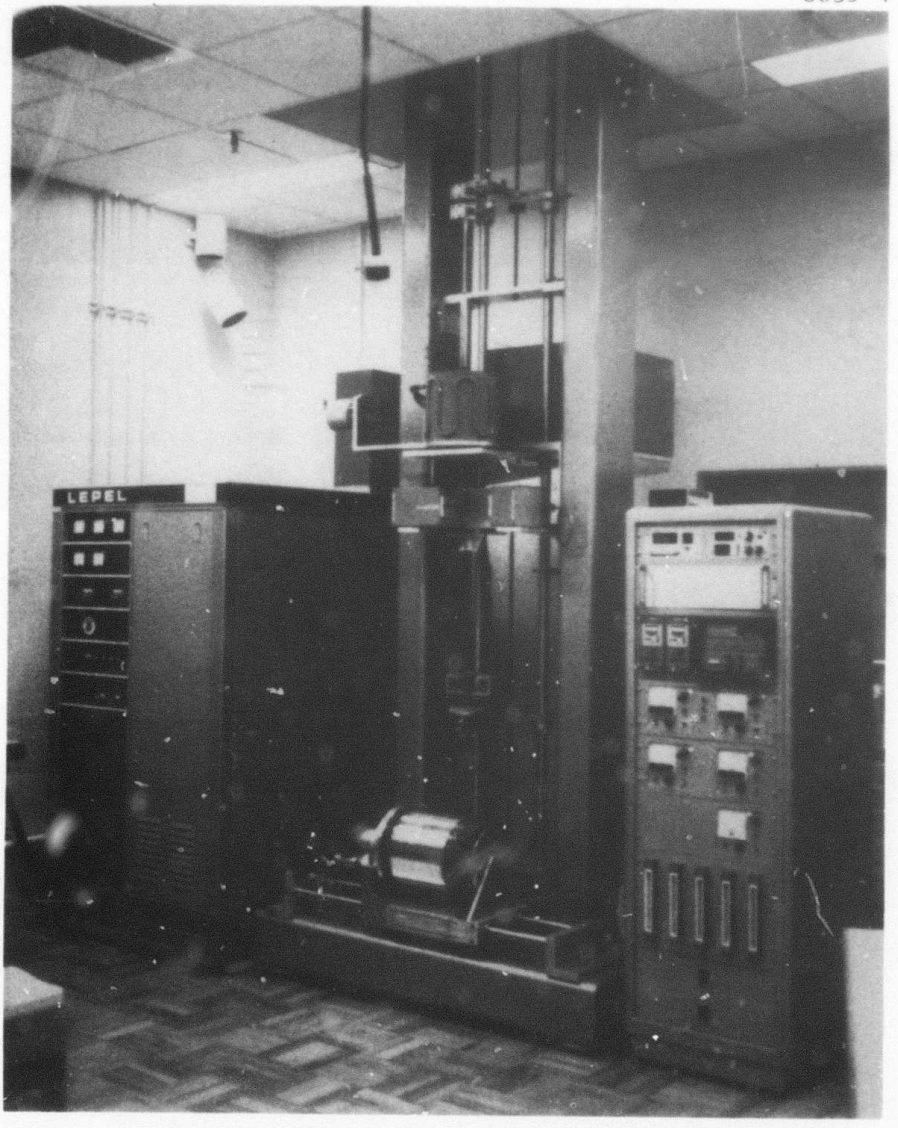

Figure 4. New fiber-drawing facility. The induction-heated drawing furnace can be operated with oxidizing, neutral, or reducing gas environments around the preform.

1. Tensile Test Procedure

The new tensile test fixture, which was described in detail in the Second Quarterly Report, was received and then tested using borosilicate glass specimens. Borosilicate glass was selected because of its ready availability and high workability (i.e., specimens were obtained without much delay). Except for the oblate bubble, these specimens had the same configuration shown in Figure 24 of the Second Quarterly Report; they were fabricated with a length of 120 mm and a diameter of ∿9.5 mm necked down to ·4.0 mm in the central portion. These specimens were coated with silicone rubber (Sylgard 182) prior to being placed in the tensile fixture. With the first two specimens, no further precautions were taken, and both of these broke at one of the flared ends. A rubber washer was slipped over the flared ends of the other specimens before they were placed in the fixture. All of the speimens protected in this way had fracture origins within the necked-down region. These specimens had not received special handling, and the wide variation in breaking stresses (from 7070 to 18,181 psi) reflects this fact. The results of these tests are summarized in Table 2. Of course, the strength results as such were not of interest, but these tests with borosilicate glass specimens showed that the new tensile test fixture was of a satisfactory design and hence that this task has been completed.

2. Specimens

Making an oblate bubble specimen first requires necking down a capillary tube in two places to isolate a small bubble. Then the region containing the bubble is reheated to allow the bubble to become spherical. Finally, the rod is pushed from both ends, which causes the bubble to become oblate. During this last step, glass flows outward causing a bulge in the region where the bubble is located. This is the same region that should have a reduced diameter compared to the rest of the specimen; therefore, it is necessary to grind this central section to the appropriate size.

Table 2. Tensile Test of Borosilicate Glass Specimens

Specimen	Load, lb	Breaking Stress, psi	Remarks
1	125	6313	Silicone rubber only Fracture at flare
2	130	6565	Silicone rubber only Fracture at flare
3	140	7070	Silicon rubber + rubber washer Fracture at necked-down region
4	150	7575	Silicone rubber + rubber washer Fracture at necked-down region
5	195	9848	Silicone rubber + rubber washer Fracture at necked-down region
6	150	7575	Silicone rubber + rubber washer Fracture at necked-down region
7	250	12626	Silicone rubber + rubber washer Fracture at necked-down region
8	190	9895	Silicone rubber + rubber washer Fracture at necked-down region
9	360	18181	Silicone rubber + rubber washer Fracture at necked-down region

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Grinding to shape was done by mounting specimens in a lathe and using a Dumore toolpost grinder with an Alundum wheel. The wheel rotated at 1750 rpm while the specimen rotated slowly in the opposite direction. A constant spray of coolant was directed at the specimen during this operation. This procedure is easy to control and provides specimens of the desired shape reproducibly.

Of course, this grinding leaves sub-surface flaws that must be removed. This is done by further grinding using SiC powder of several grit sizes, starting with 120, proceeding to 240, and finishing with 400. This is followed by polishing with 1 μm diamond paste. After mechanical polishing, the specimens are acid polished using a 15% HF-15% H_2SO_4 mixture.

Several strengths of HF as well as the 15% HF-15% H_2SO_4 mixture were tested:

- 48% HF
- 40% HF
- 24% HF

It was desirable to have a high rate of material removal together with high surface quality (i.e., surface smoothness). As expected, 48% HF provided a high etching rate (about 72 μm/hr), but it left a pebble-like surface. The etching rates of 24% HF and the 15% HF-15% H_2SO_4 mixture were similar, but the HF-H_2SO_4 mixture gave a smooth surface. The rate of removal of this 15% HF-15% H_2SO_4 mixture is $\sim$12 μm/hr if the mixture is fresh and at room temperature.

The procedure adopted was first to wash the specimens in freshly prepared chromic acid for 15 min, rinse in water, and then etch for 8 hr. The etching solution was continually kept circulating by using a magnetic stirrer. This acid treatment removed about 100 μm of surface.

Since some etch pits could usually be seen after the acid polishing, we decided to follow the HF treatment with fire polishing. The fire polishing was determined to have no effect on the size or shape of the oblate bubble. This is important, because bubbles are measured prior to polishing. Immediately after fire polishing, the specimens were coated with silicone rubber to protect the surface from mechanical damage during subsequent handling.

The flared ends of the fused silica specimens had been made by heating an end and pushing a conical tool into the open capillary while the specimen was being rotated. The flares made in this way proved unsatisfactory (as discussed in the next section). An alternate technique, suggested by the glass blower at Hughes Research Laboratories, has been shown to produce acceptable flares. Specimens are now being prepared using this technique, which is quite similar to the technique used to change the spherical bubble into an oblate spheroid. The capillary is heated at the appropriate section; pushing from both ends forms a bulge. The bulge is then cut through, leaving a flare. If specimens are properly flared and if the polishing has been done with care, the specimens should be suitable. This task seems to have been completed, but tensile tests to confirm this are required.

Table 3 lists the bubble measurements of fused silica specimens prepared since the Second Quarterly Report.

3. Fracture Initiation

The fused silica specimens were not flared as much as the borosilicate glass specimens described earlier. As a consequence, the rubber washers tended to be squeezed out of the conical section of the fixture. Various other materials were tried, but satisfactory results were not obtained. Plastic tubing, which shrinks when heated, was not pulled off of the specimens, but it proved to be too hard. The metal cut through the plastic, came into contact with the glass, and caused fracture at the flare.

Table 3. Measurements of Oblate Bubbles

Sample No.	Major Axis, mm	Minor Axis, mm
24	1.80	0.35
25	2.05	0.29
26	1.88	0.39
27	2.25	0.28
28	2.10	0.34
29	1.30	0.18
30	2.05	0.35
31	1.50	0.21
32	1.44	0.36
33	1.54	0.26
34	1.30	0.20
35	1.86	0.35
36	1.40	0.31
37	1.30	0.21
38	1.86	0.36

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It was found that bonding the rubber washers to the fused silica with a cyanoacrylate adhesive prevented slipping. One specimen treated in this way did break as intended in the necked-down region of the specimen. In fact, the fracture passed directly through the oblate bubble, but the fracture origin was clearly on the outside surface. This specimen failed at 8,000 psi; the low breaking stress was due to deep etch pits on the surface. It should be pointed out that this specimen had not been fire polished.

This task has not been completed. However, if the problems associated with specimen fabrication have been solved, it should be completed quite soon. Fused silica specimens with wider flares are being prepared and will be tested as soon as they are received from the glassblower.

SECTION 3

PLANS FOR THE NEXT QUARTER

Work will continue on all tasks discussed in Section 2. Specifically, we expect to have reportable results in the following areas:

- Optical evaluation of Al-clad waveguide with an NA of about 0.3 to ascertain the effect of this higher NA on both jacket- and axial-strain-induced attenuation
- Initial experiments to study temperature-related changes in flaw-size distribution
- Continued investigation of processing parameters on tensile strength distributions
- Initial fiber-drawing experiments with all-synthetic-glass preforms
- Static fatigue measurements (at UCLA) on doped silica specimens by the oblate bubble technique.